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Results for Method Development and Analytical Services for Water Samples from the Great Salt Lake

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SECTION 1

DETERMINATION OF Al AND OTHER TRACE ELEMENTS IN GREAT SALT LAKE USING ICP-DRC-MS

We have designed an experiment to determine the concentration of Al in Great Salt Lake using our state-of-the-art Inductively coupled plasma mass spectrometer instrument equipped with a dynamic reaction cell. This device can eliminate matrix interferences and therefore provide lower detection limits than conventional instruments. This reaction cell between the ion optics and the mass analyzer quadrupole is filled with a reactive gas such as NH₃ during analysis. Since the reaction gas reacts only with the interfering species, most interferences are eliminated with no loss in analyte signal. In our lab, we have been using NH₃ and O₂ for the determination of a suite of trace elements but since this project focused on Al mostly, we have decided to use only NH₃.

In addition to Al, we have monitored other trace elements that could be determined in seawaters using NH₃ as the reaction gas. These elements were Pb, Se, Cr, Fe, Zn, Cu, Co, and Ni. In addition to these elements, Cd and As could also be determined with this technique (also with much lower detection limits) but they require the use of O₂ and therefore not included in this short study.

The common interferences in the determination of Al by ICP-MS is due to the formation of various isotopic combinations of CN⁺ species. In addition, inductively coupled plasmas are obtained at atmospheric pressure, and there is always the tail of the highly abundant N₂ peak. In this project we have conducted the same set of experiments using two different conditions, one conventional plasma conditions, and the other at high oxide conditions in the plasma. In conventional ICP-MS analysis, formation of oxides in the plasma causes false signals for some elements such as Cd (due to MoO), and Ni, Zn, Co (due to CaO). For some elements that do not form oxides or are not affected by them somewhat increased sensitivity can be achieved by running at high oxide levels due to higher sample introduction efficiencies. We have conducted the same set of experiments at two different nebulizer gas flows representing two different oxide levels and therefore different sensitivities.

In order to determine the best analysis scheme for Al, we have analyzed the provided GSL sample at 3 different dilutions (1:10, 1:25, and 1:50). For each dilution, matrix spikes were also prepared. All results including Reference Materials (NIST 1640 and TMDW) are presented in the below tables.

Because of the much higher dissolved solids content of the Great Salt Lake sample, signal depression was observed at all dilutions. The signal depression was much more evident at the 1:10 level (~50%). The signal depression requires the choice of the right internal standard in order to correct for this effect. We have reprocessed the data with three different internal standards: Rh, Ga, and Y. It was found that Ga is a more appropriate internal standard that produced better recoveries for the spikes. As a result, Ga was used as the internal for all analytes measured except Pb and Se for which Y was used.

It was also determined that higher sensitivity at higher nebulizer gas flow produced better sensitivity without causing interference issues as determined by similar results for all analytes at each dilution.

At 25 and 50 fold dilutions, the concentrations of most analytes including Al were very close to the detection limits which resulted in high relative standard deviations. On the other hand, at the 10 fold dilution level, the recoveries of the spikes were not 100% but still within our general QA/QC limits of $\pm 25\%$ even for a difficult matrix like the Great Salt Lake water. This could be due to the fact that the spike concentration was too close to the native concentration. Even though the 10-fold dilution is harsh to the instrument, for a small batch of samples, it should be a suitable for the suite of elements monitored in this research study.

I would like to add that in this study, we didn't look at As and Cd since they require the use of O₂ as the reaction gas. For seawaters (1:10 dilution), we have determined MDL values of 130 and 5 ng/L for As and Cd, respectively. Therefore, these elements could be added to the suite if requested.

Table 1 Results of the Reference Materials with ICP-DRC-MS.

NIST 1640 (mg/L)

Analyte	m/z	Average	%RSD	TV	%Recovery
Al	27	55.6	4	52.0	107
Pb	208	29.2	2	27.9	105
Se	82	23.9	2	22.0	109
Cr	52	39.5	1	38.6	102
Fe	54	35.5	2	34.3	103
Cu	65	91.3	0.5	85.2	107
Co	59	21.8	0.1	20.3	107
Ni	58	28.8	0.3	27.4	105
Zn	64	55.8	2	53.2	105

n=3

TMDW (mg/L)

Analyte	m/z	Average	%RSD	TV	%Recovery
Al	27	129	1	120	107
Pb	208	39.5	1	40.0	99
Se	82	9.4	1	10.0	94
Cr	52	20.4	1	20.0	102
Fe	54	99.4	0.3	100.0	99
Cu	65	19.8	2	20.0	99
Co	59	25.1	1	25.0	100
Ni	58	58.8	1	60.0	98
Zn	64	72.2	1	70.0	103

n=3

Table 2 Method detection limits for GSL waters

MDL (in mg/L)

		No Dil.	10X	25X	50X
Al	27	0.030	0.300	0.751	1.502
Pb	208	0.006	0.056	0.141	0.282
Se	82	0.011	0.113	0.282	0.564
Cr	52	0.016	0.164	0.410	0.819
Fe	54	0.010	0.097	0.243	0.486
Cu	65	0.014	0.135	0.338	0.676
Co	59	0.003	0.034	0.086	0.171
Ni	58	0.002	0.023	0.057	0.113
Zn	64	0.002	0.021	0.053	0.106

Table 3 Analytical Results and Spike Recoveries for GSL water

		Analyte	m/z	Average %RSD	Spike	Measured	%RSD	% Recovery
1:50	Al	27	3.088	13	12.5	15.29	9	98
	Pb	208	1.163	2	12.5	12.60	7	91
	Se	82	0.773	11	12.5	12.58	3	94
	Cr	52	0.350	28	12.5	13.20	1	103
	Fe	54	3.944	13	12.5	16.59	1	101
	Cu	65	4.850	2	12.5	15.44	2	85
	Co	59	0.871	8	12.5	13.54	3	101
	Ni	58	1.472	11	12.5	12.73	2	90
	Zn	64	2.240	12	12.5	12.17	4	79
				n=3	n=3			

		Analyte	m/z	Average %RSD	Spike	Measured	%RSD	% Recovery
1:25	Al	27	2.684	55	6.25	7.491	7	77
	Pb	208	1.250	8	6.25	6.410	1	83
	Se	82	1.096	10	6.25	6.041	2	79
	Cr	52	0.255	5	6.25	6.052	1	93
	Fe	54	2.954	19	6.25	8.133	5	83
	Cu	65	5.508	14	6.25	9.543	1	65
	Co	59	0.837	3	6.25	6.697	2	94
	Ni	58	1.366	11	6.25	6.258	2	78
	Zn	64	2.626	26	6.25	6.105	3	56
				n=3	n=3			

		Analyte	m/z	Average %RSD	Spike	Measured	%RSD	% Recovery
1:10	Al	27	2.390	1	2.5	4.346	10	78
	Pb	208	0.995	6	2.5	3.554	4	102
	Se	82	0.970	11	2.5	2.941	4	79
	Cr	52	0.177	3	2.5	2.228	2	82
	Fe	54	1.739	9	2.5	4.329	15	104
	Cu	65	4.341	2	2.5	6.138	3	72
	Co	59	0.820	3	2.5	2.892	2	83
	Ni	58	1.074	3	2.5	2.737	3	67
	Zn	64	2.020	2	2.5	3.394	10	55
				n=3	n=3			

Section 2

Method Development for Reductive Precipitation of Great Salt Lake water samples

The reductive precipitation technique was applied to this matrix specifically for the determination of zinc. The method does not work for aluminum and Frontier Geosciences has other standard methods in place for the determination of the other metals requested by the client. Other metals were simultaneously determined by this method, and the data for all applicable metals are included in this report for comparison.

The samples were composited, filtered, split, and preserved to 0.099% nitric acid. Aliquots of the preserved sample were prepared by reductive precipitation. The reductive precipitation procedure entails the concentration of the metals of interest through selective precipitation, co-precipitation, and chelation, followed by separation from the matrix by filtration. The aqueous matrix is disposed of while the metals that were collected on the filter are redissolved in a relatively simple acidic matrix that is amenable to analysis by standard methodologies. This procedure serves not only to eliminate the prohibitive sample matrix, but it also lowers detection limits by concentrating the analytes into a smaller volume than that of the original sample.

Three sample sizes (200 mL, 100 mL, and 50 mL) were tested in anticipation of any difficulties that might result from the very high dissolved solids content of the water. Samples and matrix spikes were prepared in triplicate for each sample size. The spiking concentrations were tailored to the samples in order to verify the ability to quantitate at the levels indigenous to the matrix. The sample batch also included three preparation blanks, three spiked blanks, and a Certified Reference Material (CASS-4). The prepared samples were analyzed by ICP-MS. Table 4 summarizes the QC data for the analytical batch.

Table 4. QC data for Great Salt Lake sample batch

Analyte	Isotope	MDL	Preparation Blanks Mean* µg/L	CASS-4			Spike µg/L	Spike Blanks Recovery	
				T.V. µg/L	Measured µg/L	Recovery %R		Mean* %R	RSD %
Zinc	⁶⁶ Zn	0.076	0.097	0.381	0.28	72.8%	2.5	74.7%	20.1%
Arsenic	⁷⁵ As	0.009	<0.009	1.110	0.89	79.9%	25.0	78.9%	1.2%
Cadmium	¹¹⁴ Cd	0.004	<0.004	0.026	0.034	129% [§]	0.10	82.2%	6.2%
Copper	⁶⁵ Cu	0.064	0.069	0.592	0.51	85.5%	5.0	76.9%	2.5%
Lead	^{206,207,208} Pb	0.013	0.043	0.010	<0.013		1.0	96.1%	3.4%
Nickel	⁶⁰ Ni	0.033	<0.033	0.314	0.26	82.7%	1.0	76.3%	1.7%
Selenium	⁷⁷ Se	0.022	<0.022	NC	<0.022		0.50	68.7%	5.6%

* n=3

[§] Mo interference on Cd

NC = not certified

As noted in the table, the high recovery for cadmium in CASS-4 may be due to an interference from molybdenum. The reductive precipitation sample matrix makes it difficult to compensate for this interference through the use of standard ICP-MS interelement corrections. Molybdenum was found to be in the Great Salt Lake (>100 µg/L in the concentrate) in sufficient quantities to warrant concern about a positive bias on the cadmium data.

Tables 5 through 7 show the analytical and QC results for the three sizes of Great Salt Lake samples employed in this study.

Table 5. QC data for Great Salt Lake samples (50 mL sample)

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Spike	Matrix Spikes	
			Mean*	RSD		Mean Recovery*	RSD
			$\mu\text{g/L}$	%	$\mu\text{g/L}$	%R	%
Zinc	⁶⁶ Zn	0.30	2.70	27.9%	0.75	42.7%	5.2%
Arsenic	⁷⁵ As	0.038	80.9	4.6%	7.5	54.0%	20.0%
Cadmium	¹¹⁴ Cd	0.015	0.081	7.2%	0.030	79.1%	5.2%
Copper	⁶⁵ Cu	0.25	5.41	24.5%	1.5	63.6%	4.7%
Lead	^{206,207,208} Pb	0.050	1.13	9.6%	0.30	87.6%	6.0%
Nickel	⁶⁰ Ni	0.13	1.02	7.0%	0.30	76.7%	2.2%
Selenium	⁷⁷ Se	0.090	<0.090	3.3%	0.15	70.2%	2.2%

Arsenic over range * n=3

Table 6. QC data for Great Salt Lake samples (100 mL sample)

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Spike	Matrix Spikes	
			Mean*	RSD		Mean Recovery*	RSD
			$\mu\text{g/L}$	%	$\mu\text{g/L}$	%R	%
Zinc	⁶⁶ Zn	0.15	1.84	2.9%	1.3	75.3%	8.0%
Arsenic	⁷⁵ As	0.019	70.9	2.8%	12.5	93.4%	3.5%
Cadmium	¹¹⁴ Cd	0.007	0.081	1.1%	0.05	79.8%	4.7%
Copper	⁶⁵ Cu	0.13	4.32	1.5%	12.5	76.4%	1.9%
Lead	^{206,207,208} Pb	0.025	1.05	2.4%	0.5	96.5%	3.3%
Nickel	⁶⁰ Ni	0.067	0.93	1.5%	0.5	76.3%	0.4%
Selenium	⁷⁷ Se	0.045	0.079	4.5%	0.25	67.3%	4.2%

Arsenic over range * n=3

Table 7. QC data for Great Salt Lake samples (200 mL sample)

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Spike	Matrix Spikes	
			Mean*	RSD		Mean Recovery*	RSD
			$\mu\text{g/L}$	%	$\mu\text{g/L}$	%R	%
Zinc	⁶⁶ Zn	0.076	2.10	0.5%	2.5	67.4%	3.0%
Arsenic	⁷⁵ As	0.009	75.4	2.7%	25.0	68.5%	3.7%
Cadmium	¹¹⁴ Cd	0.004	0.072	3.8%	0.10	83.6%	2.5%
Copper	⁶⁵ Cu	0.064	4.29	2.6%	5.0	76.3%	2.9%
Lead	^{206,207,208} Pb	0.013	1.09	2.5%	1.0	97.7%	3.4%
Nickel	⁶⁰ Ni	0.033	0.91	2.6%	1.0	73.6%	2.7%
Selenium	⁷⁷ Se	0.022	0.046	16.1%	0.50	40.8%	38.5%

Arsenic over range * n=3

The smallest sample size (50 mL) exhibited some of the poorest precision. While the aliquot size was adequate to detect zinc and most of the other elements presented in Table 2, the low

concentrations were evidently susceptible to contamination. The effect is only exaggerated with the use of large correction factors used to account for sample pre-concentration.

The intermediate sample size (100 mL) presented no particular difficulties. The data in Table 3 illustrate the overall best performance of this method for most of the metals of interest.

The largest sample size (200 mL) presented a number of experimental difficulties due to the high level of dissolved solids in the samples. While the analytical performance (Table 4) was not gravely affected, there were physical problems in the preparation and analysis. There was a great deal of precipitate. This in itself suggests that calcium and magnesium were precipitating in some great quantity. This reduces the degree of separation of analytes from matrix and it slows the filtration. Furthermore, extra rinsing becomes necessary in order to eliminate as much of the precipitated matrix as possible. However the filters become so thoroughly clogged with these samples that rinsing becomes impractical. Furthermore, some of the precipitate was always lost through adherence to the walls and neck of the filter funnel.

The sheer quantity of precipitate caused problems for the digestion step as well. A good deal of undissolved matter remained after the digestion procedure was completed. The method performance for this sample size is comparable to that achieved with the intermediate sample size, but the presence of undissolved material in the digest poses a hazard to the analytical system because of the possibility that particulate matter may clog the ICP-MS sample introduction system.

Section 3

Analysis of Great Salt Lake samples by ICP-MS following extraction of Cd, Cu, Pb and Ni by Co-APDC Co- precipitation.

A sample of water from the Great Salt Lake Utah was received and filtered through a 0.45um Gelman type in-line filter. The filtrate was then preserved to 0.099% nitric acid. The sample was prepared for analysis on a Perkin-Elmer ELAN 6000 ICP-MS system using a quantitative extraction/ preconcentration technique which allows for the simultaneous co-precipitation of lead, copper, cadmium and nickel from seawater and other difficult matrices. This procedure serves not only to eliminate the prohibitive sample matrix, but it also lowers detection limits by concentrating the analytes into a smaller volume than that of the original sample

The sample was prepared in duplicate using a variety of aliquot sizes in anticipation of any possible issues arising from the high dissolved solid in the sample. Matrix spikes were prepared at the same time at three different concentrations allowing for spike recoveries and a method of standard additions to be calculated, for each aliquot size.

The digestion batch (in addition to matrix QC) included:

- Four preparation blanks
- One blank spike
- Two certified reference materials (including CASS-4, a seawater CRM)

The maximum volume of sample which can be concentrated, by the method, is 200ml. When less sample volume is used, the final volume is raised to 200ml by the addition of ultra pure milli-Q (reagent water). This method is particularly prone to fluctuations in the pH of the sample. The final aliquot should have a pH of 1.9 for maximum extraction efficiency. Samples have their pH adjusted to reflect this requirement.

The sample in question was prepared using the following aliquot sizes: 200ml, 100ml, 50ml, 25ml, 10ml, and 5ml. Once the samples have been brought to 200ml (if using less than 200ml sample) and pH adjusted, 1ml of both cobalt (II) solution and APDC (the complexing agent) was added. This forms a precipitate. The sample was capped and left overnight. The precipitate was collected the following day by filtration and re-dissolved in concentrated nitric acid, then diluted in 5.0 % HNO₃. Analysis is performed by ICP-MS using online internal standardization and robust plasma conditions.

A number of issues arose with this digestion batch. The 200ml and 100ml aliquots proved difficult to filter due to the clogging of the filters by a large amount of precipitate. Recovery in the blank spikes and reference materials also proved unreliable. The poor recoveries are possibly the result of using a partly degraded complexing agent or the presence of organics in the water supply. These issues were addressed and a second digestion was performed.

The second batch comprised:

- Four preparation blanks
- One blank spike
- Two certified reference materials (including CASS-4, a seawater CRM)
- The sample prepared in duplicate using two aliquot sizes (50ml and 25ml)
- Matrix spike and spike duplicates for each aliquot size

Results for both digestion batches are given below.

Table 8 QC Data for Digestion Batch 1

Analyte	Isotope	MDL	Preparation Blanks Mean µg/L	CASS-4			Spike µg/L	Blank Spike Mean* %Rec
				T.V. µg/L	Measured µg/L	Recovery %Rec		
Cadmium	¹¹⁴ Cd	0.0133	0.0049	0.026	0.048	184.7%	0.250	3809%
Copper	⁶⁵ Cu	0.0402	0.1635	0.592	0.495	83.6%	3.125	96.8%
Lead	²⁰⁸ Pb	0.0174	0.0174	0.010	4.936	50367	0.625	66.5%
Nickel	⁶⁰ Ni	0.0412	0.1730	0.314	0.2658	84.6%	3.125	66.5%

Table 9. QC data for Great Salt Lake samples (200 mL sample). Batch 1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope (av % rec)	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.0027	0.1299	15.1	0.4047	0.9991	0.3635
Copper	⁶⁵ Cu	0.0080	5.178	2.5	0.8072	0.9998	5.838
Lead	²⁰⁸ Pb	0.0035	0.6165	1.1	0.5103	0.9605	0.6404
Nickel	⁶⁰ Ni	0.0082	1.238	6.3	0.6552	0.9999	1.348

Table 10. QC data for Great Salt Lake samples (100 mL sample). Batch1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope (av % rec)	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.0053	0.1399	6.8	0.5108	0.9960	0.2698
Copper	⁶⁵ Cu	0.0161	4.830	10.3	0.9397	0.9992	5.114
Lead	²⁰⁸ Pb	0.0070	0.7388	6.8	0.7274	0.9944	1.002
Nickel	⁶⁰ Ni	0.0165	1.207	29.2	0.6959	0.9994	1.473

Table 11. QC data for Great Salt Lake samples (50 mL sample). Batch 1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.0106	0.1502	0.7	0.6739	0.994	0.2387
Copper	⁶⁵ Cu	0.0322	5.319	4.8	0.8893	0.9987	6.329
Lead	²⁰⁸ Pb	0.014	0.8286	4.0	0.7091	0.9603	1.222
Nickel	⁶⁰ Ni	0.0330	1.739	0.8	0.7371	0.999	2.525

Table 12. QC data for Great Salt Lake samples (25 mL sample). Batch 1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope (av % rec)	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.0177	0.1310	11.1	0.5272	0.9974	0.2551
Copper	⁶⁵ Cu	0.0536	5.204	6.7	0.7907	0.9969	6.1763
Lead	²⁰⁸ Pb	0.0233	0.8624	3.9	0.4695	0.8291	1.762
Nickel	⁶⁰ Ni	0.0550	2.432	7.2	0.5762	0.9999	4.014

Table 13. QC data for Great Salt Lake samples (10 mL sample). Batch 1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope (av % rec)	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.0531	0.1540	5.6	0.5967	0.9855	0.3146
Copper	⁶⁵ Cu	0.1609	9.251	4.2	0.8895	0.9989	11.08
Lead	²⁰⁸ Pb	0.0698	1.333	2.0	0.5476	0.8203	2.642
Nickel	⁶⁰ Ni	0.1650	6.017	2.6	0.6305	0.9995	9.966

Table 14. QC data for Great Salt Lake samples (5 mL sample). Batch 1

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Matrix Spikes		
			Mean (µg/L)	%RSD	Slope (av % rec)	Correlation coefficient	Intercept
Cadmium	¹¹⁴ Cd	0.1062	0.1824	2.2	0.7850	0.998	0.2210
Copper	⁶⁵ Cu	0.3218	12.92	12.1	0.6323	0.3092	28.80
Lead	²⁰⁸ Pb	0.1395	1.996	6.6	0.6254	0.7332	3.146
Nickel	⁶⁰ Ni	0.3299	12.07	0.8	0.5418	0.3633	29.55

Table 15 QC Data for Digestion Batch 2

Analyte	Isotope	MDL	Preparation Blanks Mean µg/L	CASS-4			Blank Spike	
				T.V. µg/L	Measured µg/L	Recovery %R	Spike µg/L	Mean* %R
Cadmium	¹¹⁴ Cd	0.006	0.006	0.026	0.0386	148.6	0.25	66.2
Copper	⁶⁵ Cu	0.0910	0.1226	0.592	0.4551	76.9	3.125	78.1
Lead	²⁰⁸ Pb	0.0149	0.0122	0.0154	0.0081	53.3	0.625	74.9
Nickel	⁶⁰ Ni	0.0512	0.0929	0.314	0.2294	73.0	3.125	56.6

Table 16. QC data for Great Salt Lake samples (50 mL sample). Batch 2

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Spike (µg/L)	Matrix Spikes	
			Mean (ug/L)	% RPD		Mean Recovery %	%RPD
Cadmium	¹¹⁴ Cd	0.0091	0.0525	66.2	1	77.1	3.7
Copper	⁶⁵ Cu	1.4555	3.4312	88.8	10	83.0	4.1
Lead	²⁰⁸ Pb	0.2382	0.6118	88.6	2	99.6	1.8
Nickel	⁶⁰ Ni	0.8184	2.2631	20.4	10	83.2	6.6

Table 17. QC data for Great Salt Lake samples (25 mL sample). Batch2

Analyte	Isotope	MDL	Great Salt Lake				
			Sample Concentration		Spike (µg/L)	Matrix Spikes	
			Mean (µg/L)	% RPD		Mean Recovery %	%RPD
Cadmium	¹¹⁴ Cd	0.0182	0.0820	2.5	1	107.8	5.5
Copper	⁶⁵ Cu	2.9110	4.4539	7.8	10	153.4	2.6
Lead	²⁰⁸ Pb	0.4763	0.9673	0.7	2	146.8	0.9
Nickel	⁶⁰ Ni	1.6368	1.8138	37.9	10	130.4	8.4

The results suggest that the best aliquot size to use for the extraction and analysis of this type of sample would be 50 mLs.

Section 4
ANALYSIS OF GREAT SALT LAKE WATER FOR Se BY HYDRIDE
GENERATION ATOMIC FLOURESCENCE SPECTROSCOPY

A sample of water from the Great Salt Lake Utah was received and filtered through a 0.45um Gelman type in-line filter. The filtrate was then preserved to 0.099% nitric acid. The sample was prepared for analysis on a PSA Millennium Excalibur atomic fluorescence spectroscopy system. The sample preparation involves the conversion of all selenium present in the samples to selenium IV. Selenium IV forms a volatile hydride, which can be quantitatively measured in the low part per trillion range.

The sample was run in duplicate using two aliquot sizes 2ml (10x) and 4ml (5x) to determine the most suitable dilution factor and thus detection limit. Matrix spikes were prepared at the same time at three different concentrations allowing for spike recoveries and a method of standard additions to be calculated, for each aliquot size.

The digestion batch (in addition to matrix QC) included:

- Four digestion blanks
- Two laboratory controlled water standards
- Two certified reference materials

It was decided to not run the samples less than 5x because of the physical effects of the high salt concentration on the instruments capabilities.

The results with a five fold dilution are acceptable and within all QA requirements. I suggest that samples analyzed by this method be run at this dilution.

Results for all aliquot sizes are given below.

Table 16 - Sample Results for Se in GSL Waters

Sample ID	Total Se (ng/L)
Great salt lake water	575.7

Table 17 - QC Sample Summary for Se in GSL Waters

Method Blanks	Total Se (ng/L)
DBW1	16.66
DBW2	12.68
DBW3	26.66
DBW4	7.741
Mean	15.93
Std. Dev.	8.025
Est. MDL	24.08

Table 18 - QC Sample Summary for Se in GSL Waters (continued)

Standard Ref. Material	Total Se (ng/L)
Source	HP CRM-SW
Cert. Value	4000
Obs. Value	4238
% Rec.	106.0%

Matrix QC Sample	Total Se (ng/L)	Matrix QC Sample	Total Se (ng/L)
Sample ID	Great salt lake water	Sample ID	Great salt lake water
Dilution	10	Dilution	5
Sample Conc.	742.3	Sample Conc.	575.7
MD Conc.	722.8	MD Conc.	630.3
RPD	2.7	RPD	9.1
MS Spiking Level	1250	MS Spiking Level	625
MS Conc.	1831	MS Conc.	1230
MS % Rec.	87.9	MS % Rec.	100.4
MSD Spiking Level	2500	MSD Spiking Level	1250
MSD Conc.	3083	MSD Conc.	1788
MSD % Rec.	94.0	MSD % Rec.	94.8
MST Spiking Level	3750	MST Spiking Level	1875
MST Conc.	3932	MST Conc.	2332
MST % Rec.	85.3	MST % Rec.	92.2
MSA Slope	0.868	MSA Slope	0.919
MSA Correlation coefficient	0.997	MSA Correlation coefficient	0.999